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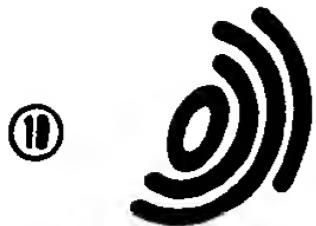
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④ Treating a temperature-sensitive hydrocarbonaceous waste stream containing a non-distillable component.

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Description

This invention relates to the production of a distillable hydrocarbonaceous product from a temperature-sensitive hydrocarbonaceous waste stream containing a non-distillable component. More specifically, the invention relates to a process using flash vaporization for treating a temperature-sensitive hydrocarbonaceous waste stream containing a non-distillable component to produce a distillable hydrocarbonaceous product and a heavy product comprising a non-distillable component, while minimizing thermal degradation of the hydrocarbonaceous stream.

With the advent of recognition of the dangers associated with disposal of waste streams containing hazardous materials, there has been a steadily increasing demand for technology which is capable of treating a temperature-sensitive hydrocarbonaceous waste stream containing a non-distillable component to produce a distillable hydrocarbonaceous product and a heavy non-distillable product, while minimizing thermal degradation of the hydrocarbonaceous feed stream. Such treatment has always been in demand for the preparation and production of various hydrocarbonaceous products, but with the increased environmental emphasis for the treatment and recycle of waste hydrocarbonaceous products, there is an increased need for improved processes to separate heavy non-distillable components from a distillable hydrocarbonaceous product. For example, during the disposal or recycle of potentially environmentally harmful hydrocarbonaceous waste streams, an important step in the total solution to the problem, is the pretreatment or conditioning of a hydrocarbonaceous stream which facilitates the ultimate resolution to provide product streams which may subsequently be handled in an environmentally acceptable manner. Therefore, those skilled in the art have sought to find feasible techniques to remove heavy non-distillable components from a temperature-sensitive hydrocarbonaceous waste stream to provide a distillable hydrocarbonaceous product. Previous techniques which have been employed include filtration, vacuum wiped-film evaporation, solvent extraction, centrifugation, and vacuum distillation.

The invention provides an improved process for the production of a distillable hydrocarbonaceous product from a temperature-sensitive hydrocarbonaceous waste stream containing a non-distillable component by means of contacting the hydrocarbonaceous waste feed stream with a hot hydrogen-rich gaseous stream under conditions selected to increase the temperature of the feed stream and to flash vaporize at least a portion of the distillable hydrocarbonaceous compounds, thereby producing a distillable hydrocarbonaceous product. Important elements of the improved process are the relatively short time that the feed stream is maintained at elevated temperature, the avoidance of heating the feed stream via indirect heat exchange, and the inhibition of coking reactions which results from the presence of hydrogen.

One embodiment of the invention may be characterized as a process for treating a temperature-sensitive hydrocarbonaceous waste stream containing a non-distillable component to produce a distillable hydrocarbonaceous product and a heavy product comprising the non-distillable component while minimizing thermal degradation of the hydrocarbonaceous stream which process comprises the steps of:

- (a) contacting the hydrocarbonaceous stream with a hot first hydrogen-rich gaseous stream having a temperature greater than the hydrocarbonaceous stream in a flash zone at flash conditions thereby increasing the temperature of the hydrocarbonaceous stream and vaporizing at least a portion thereof to provide a vapor stream comprising hydrogen and hydrocarbons and a heavy product comprising the non-distillable component;
- (b) condensing at least a portion of the vapor stream to provide a second hydrogen-rich gaseous stream suitable for recycle and a liquid stream comprising fuel gas and distillable hydrocarbonaceous compounds; and
- (c) recovering a distillable hydrocarbonaceous product from the liquid stream.

Another embodiment of the invention may be characterized as a process for treating a temperature-sensitive hydrocarbonaceous waste stream containing a non-distillable component to produce a distillable hydrocarbonaceous product and a heavy product comprising the non-distillable component while minimizing thermal degradation of the hydrocarbonaceous stream which process comprises the steps of:

- (a) contacting the hydrocarbonaceous waste stream with a hot first hydrogen-rich gaseous stream having a temperature greater than the hydrocarbonaceous stream in a flash zone at flash conditions thereby increasing the temperature of the hydrocarbonaceous stream and vaporizing at least a portion thereof to provide a first vapor stream comprising hydrogen and hydrocarbons and a heavy product comprising the non-distillable product;
- (b) condensing at least a portion of the first vapor stream to provide a second hydrogen-rich gaseous stream suitable for recycle and a liquid stream comprising distillable hydrocarbonaceous compounds and dissolved hydrogen;
- (c) separating the liquid stream to provide a fuel gas stream and a normally liquid distillable hydrocarbonaceous product, and
- (d) heating at least a portion of said second hydrogen-rich gaseous stream, and passing it to step (a) as at least a portion of said first hydrogen-rich gaseous stream.

Other embodiments of the present invention encompass further details such as preferred feedstocks and operating conditions, all of which are hereinafter disclosed in the following discussion of each of these facets of the invention.

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The drawing is a simplified process flow diagram of a preferred embodiment of the present invention.

The present invention provides an improved process for the removal of heavy non-distillable components from a temperature-sensitive hydrocarbonaceous stream. A wide variety of temperature-sensitive hydrocarbonaceous streams may be employed for feed streams treated in accordance with the process of the present invention. Examples of hydrocarbonaceous streams which are suitable for treatment by the process of the present invention are dielectric fluids, hydraulic fluids, heat transfer fluids, used lubricating oil, used cutting oils, used solvents, still bottoms from solvent recycle operations, coal tars, atmospheric residuum, oils contaminated with polychlorinated biphenyls (PCB), pesticide wastes or other hydrocarbonaceous industrial waste. Many of these hydrocarbonaceous streams may contain non-distillable components which include, for example, organometallic compounds, inorganic metallic compounds, finely divided particulate matter and non-distillable hydrocarbonaceous compounds (i.e. hydrocarbons or hydrocarbonaceous compounds boiling above the maximum boiling point for materials that can be recovered in the overhead from a vacuum fractionation column). The present invention is particularly advantageous when the non-distillable components comprise sub-micron particulate matter and the conventional techniques of filtration or centrifugation tend to be highly ineffective.

Once the temperature-sensitive hydrocarbonaceous feed stream is separated into a distillable hydrocarbonaceous product and a heavy non-distillable product, each of these products may be utilized as recovered, or may be subsequently treated or processed by any known technique or process. If the feed stream contains metallic compounds such as those that contain metals such as zinc, copper, iron, barium, phosphorus, magnesium, aluminium, lead, mercury, cadmium, cobalt, arsenic, vanadium, chromium, and nickel, these compounds will be isolated in the relatively small volume of recovered non-distillable product, which may then be treated for metals recovery or otherwise disposed of as desired. In the event that the feed stream contains distillable hydrocarbonaceous compounds which include sulphur, oxygen, nitrogen, metal or halogen components, the resulting recovered distillable hydrocarbonaceous product may be further processed to remove or convert any such components as desired or required.

In accordance with the subject invention, a temperature-sensitive hydrocarbonaceous stream containing a non-distillable component is contacted with a hot hydrogen-rich gaseous stream having a temperature greater than the hydrocarbonaceous stream in a flash zone at flash conditions, thereby increasing the temperature of the hydrocarbonaceous stream and vaporizing at least a portion thereof to provide a hydrocarbonaceous vapor stream comprising hydrogen and a heavy non-distillable product. The hydrogen-rich gaseous stream preferably comprises more than 80 mol % hydrogen and more preferably more than 90 mol % hydrogen. The hydrogen-rich gaseous stream is multi-functional and serves as 1) a heat source used for directly heating the hydrocarbonaceous feed stream so as to preclude the cok formation that could otherwise occur when using an indirect heating apparatus, such as a heater or heat-exchanger, 2) a diluent to reduce the partial pressure of the hydrocarbonaceous compounds, 3) a possible reactant to minimize the formation of hydrocarbonaceous polymers, and 4) a stripping medium. In accordance with the subject invention, the temperature-sensitive hydrocarbonaceous feed stream is preferably maintained at a temperature less than 482°F (250°C) before being introduced into the flash zone, in order to prevent or minimize the thermal degradation of the feed stream. Depending upon the characteristics and composition of the hydrocarbonaceous feed stream, the hot hydrogen-rich gaseous stream is introduced into the flash zone at a temperature greater than the hydrocarbonaceous feed stream and preferably at a temperature from 200°F (93°C) to 1200°F (649°C).

During the contacting, the flash zone is preferably maintained at flash conditions which include a temperature from 150°F (65°C) to 860°F (460°C), a pressure from atmospheric to 2000 psig (13788 kPa gauge), a hydrogen circulation rate of 1000 SCFB (168 normal m³/m³) to 30,000 SCFB (5056 normal m³/m³), based on the temperature-sensitive hydrocarbonaceous feed stream, and an average residence time of the hydrogen-containing, hydrocarbonaceous vapor stream in the flash zone from 0.1 to 50 seconds. A more preferred average residence time of the hydrogen-containing, hydrocarbonaceous vapor stream in the flash zone is from 1 to 10 seconds.

The resulting heavy non-distillable portion of the feed stream is removed from the bottom of the flash zone as required to yield a heavy non-distillable product. The heavy non-distillable product may contain a relatively small amount of distillable components but since essentially all of non-distillable components contained in the hydrocarbonaceous feed stream are recovered in this product stream, the term "heavy non-distillable product" is nevertheless used for the convenient description of this product stream. The heavy non-distillable product preferably contains an atmospheric distillable component of less than 10 weight percent and more preferably less than 5 weight percent. Under certain circumstances, with a feed stream not having an appreciable amount of liquid non-distillable components, it is contemplated that an additional liquid may be utilized to flush the heavy non-distillables from the flash zone. An example of this situation is when the hydrocarbonaceous feed stream comprises a very high percentage of distillable hydrocarbonaceous compounds and relatively small quantities of finely divided particulate matter (solid) and essentially no liquid non-distillable component for use as a carrier for the solids. Such a flush liquid may, for example, be a diesel cut boiling in the range from 500°F (260°C) to 700°F (371°C), a high boiling range vacuum gas oil having a boiling range from 700°F (371°C) to 1000°F (538°C), or a vacuum tower bottoms stream boiling at a temperature greater than 1000°F (538°C). The selection of a flush liquid depends upon the composition of the hydrocarbonaceous feed stream and the prevailing flash conditions

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in the flash separator, and the volume of the flush liquid is preferably limited to that required for removal of the heavy non-distillable component.

The resulting hydrocarbonaceous vapor stream comprising hydrogen is removed from the flash zone, and at least a portion thereof is condensed to provide a second hydrogen-rich gaseous stream and a liquid stream comprising distillable hydrocarbonaceous compounds.

In the drawing, one embodiment of the subject invention is illustrated by means of a simplified flow diagram, in which such details as pumps, instrumentation, heat-exchange and heat-recovery circuits, compressors and similar hardware have been deleted as being non-essential to an understanding of the techniques involved. The use of such miscellaneous apparatus is well known to one skilled in the art of hydrocarbon processing techniques. With reference now to the drawing, a waste oil feed stream having a non-distillable component is introduced into the process via conduit (1), and is contacted with a hot gaseous hydrogen-rich recycle stream which is provided via conduit (7) and hereinafter described. The waste oil and the hydrogen-rich recycle stream are intimately contacted in hot hydrogen flash separator (2). A hydrocarbonaceous vapor stream comprising hydrogen is removed from hot hydrogen flash separator (2) via conduit (3), cooled in heat-exchanger (4), and introduced into high pressure vapor/liquid separator (5). A heavy non-distillable stream is removed from the bottom of hot hydrogen flash separator (2) via conduit (6) and recovered. A hydrogen-rich gaseous stream is removed from separator (5) via conduit (7), heated to a suitable temperature in heat-exchanger (14), and utilized to contact the waste oil feed stream and hereinabove described. Since hydrogen is lost from the process because a portion of the hydrogen will be dissolved in the liquid hydrocarbon leaving separator (5), it is necessary to supplant the lost hydrogen with make-up hydrogen from some suitable external source, i.e., a catalytic reforming unit or a hydrogen plant. Make-up hydrogen may be introduced into the system at any convenient and suitable point, and is introduced in the embodiment shown in the drawing via conduit (8). A liquid hydrocarbonaceous stream containing hydrogen in solution is removed from high pressure vapor/liquid separator (5) via conduit (9) and is introduced into low pressure vapor/liquid separator (10). A gaseous stream comprising hydrogen and any normally gaseous hydrocarbons present is removed from low pressure vapor/liquid separator (10) via conduit (11) and recovered. A normally liquid distillable hydrocarbonaceous product is removed from low pressure vapor/liquid separator (10) via conduit (12) and recovered. In the event that the waste oil feed stream contains water, this water is recovered from low pressure vapor/liquid separator (10) via conduit (13).

The following Example is presented for the purpose of further illustrating the process of the present invention, and to indicate the benefits afforded by the utilization thereof in producing a distillable hydrocarbonaceous product, while minimizing thermal degradation of the temperature-sensitive hydrocarbonaceous feed stream containing a non-distillable component.

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Example

A waste lube oil containing fully divided particulate matter and heavy metals having the characteristics presented in Table 1, and contaminated with 1020 parts per million by weight (wppm) of polychlorinated biphenyl (PCB), in the form of Aroclor, was charged at a rate of 100 mass units per hour to a hot hydrogen flash zone. The hot hydrogen was introduced into the hot hydrogen flash zone at a rate of 31 mass units per hour.

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TABLE 1
WASTE LUBE OIL FEEDSTOCK PROPERTIES (5375—45)

| | | |
|-------|---|----------------|
| | Specific Gravity @ 60°F (15°C) | .8827 |
| 5 | Vacuum Distillation Boiling Range, (ASTM D—1160) | °F °C |
| <hr/> | | |
| 10 | IBP | 338 (170) |
| | 10% | 516 (269) |
| | 20% | 628 (331) |
| 15 | 30% | 690 (367) |
| | 40% | 730 (388) |
| 20 | 50% | 750 (399) |
| | 60% | 800 (421) |
| | 70% | 831 (444) |
| 25 | 80% | 882 (474) |
| | % Over | 80 |
| 30 | % Bottoms | 20 |
| <hr/> | | |
| | Sulphur, weight percent | 0.5 |
| 35 | Polychlorinated Biphenyl Concentration, wppm | 1020 |
| | Lead, wppm | 863 |
| | Zinc, wppm | 416 |
| 40 | Cadmium, wppm | 1 |
| | Copper, wppm | 21 |
| 45 | Chromium, wppm | 5 |

The waste lube oil was preheated to a temperature of <482°F (<250°C) before introduction into the flash zone, which temperature precluded any significant detectable thermal degradation. The waste lube oil was intimately contacted in the flash zone with a hot hydrogen-rich gaseous stream having a temperature upon introduction into the flash zone of >748°F (>398°C). In addition, the hot hydrogen flash zone was operated at conditions which included a temperature of 748°F (398°C), a pressure of 500 psig (3447 kPa gauge), a hydrogen circulation rate of 18000 SCFB (3034 normal m³/m³) and an average residence time of the vapor stream of 5 seconds. A hydrocarbonaceous vapor stream comprising hydrogen was recovered from the flash separation zone, cooled to 77°F (25°C) and introduced into a high pressure separator. An overhead gas stream containing 100% hydrogen in an amount of 31 mass units per hour was recovered from the high pressure separator and found to be suitable for recycle to the flash zone. A liquid stream was removed from the high pressure separator and introduced into a low pressure separator to provide a fuel gas stream containing hydrogen and a small amount of light hydrocarbons and a liquid bottoms stream in the amount of 88 mass units per hour having the characteristics presented in Table 2.

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TABLE 2
ANALYSIS OF LOW PRESSURE SEPARATOR BOTTOMS STREAM

| | | |
|-------------------------|---|----------------|
| 5 | Specific Gravity @ 60°F (15°C) | 0.866 |
| 10 | Vacuum Distillation Boiling Range, (ASTM D—1160) | °F (°C) |
| IBP | | |
| 15 | 10% | 225 (107) |
| 20 | 20% | 433 (223) |
| 25 | 30% | 538 (280) |
| 30 | 40% | 633 (334) |
| 35 | 50% | 702 (372) |
| 40 | 60% | 741 (394) |
| 45 | 70% | 770 (410) |
| 50 | 80% | 801 (427) |
| 55 | 90% | 837 (447) |
| 60 | 95% | 896 (479) |
| 65 | EP | 943 (506) |
| % Over | | 982 (527) |
| % Bottoms | | 97 |
| Sulphur, weight percent | | 3 |
| 40 | Polychlorinated Biphenyl Concentration, wppm | 0.31 |
| 45 | Lead, wppm | 1143 |
| 50 | Zinc, wppm | 3.7 |
| 55 | Cadmium, wppm | 1.5 |
| 60 | Copper, wppm | <0.04 |
| 65 | Chromium, wppm | 0.1 |
| | | 0.6 |

A non-distillable liquid stream was recovered from the bottom of the flash separation zone in an amount of 12 mass units per hour and having the characteristics presented in Table 3.

TABLE 3
ANALYSIS OF NON-DISTILLABLE STREAM

| | | |
|----|--|-----|
| 55 | Specific Gravity @ 60°F (15°C) | 0.9 |
| 60 | Polychlorinated Biphenyl Concentration, wppm | 110 |

In summary, this example demonstrated that a waste lube oil having a non-distillable component and containing 1020 wppm of polychlorinated biphenyl and 1306 wppm of heavy metals, i.e., lead, zinc, cadmium, copper and chromium, was separated into a distillable hydrocarbonaceous stream containing

98.6 weight percent of the polychlorinated biphenyl contained in the waste lube oil and a heavy stream comprising essentially all of the non-distillable component of the waste lube oil, including 99.5 weight percent of the heavy metals. The analysis of the overhead gas stream showed that the temperature-sensitive waste lube oil did not experience any significant amounts of undesirable thermal cracking, with 5 the accompanying formation of normally gaseous hydrocarbonaceous compounds.

Claims

- 10 1. A process for treating a temperature-sensitive hydrocarbonaceous waste stream (1) containing a non-distillable component to produce a distillable hydrocarbonaceous product and a heavy product comprising said non-distillable component while minimizing thermal degradation of said hydrocarbonaceous stream characterized by:
 - (a) contacting said hydrocarbonaceous waste stream (1) with a hot first hydrogen-rich gaseous stream (7) having a temperature greater than said hydrocarbonaceous stream, in a flash zone (2) at flash conditions, thereby increasing the temperature of said hydrocarbonaceous stream and vaporizing at least a portion thereof to provide a vapor stream (3) comprising hydrogen and hydrocarbons, and a heavy product (6) comprising said non-distillable component;
 - (b) condensing (4) at least a portion of said vapor stream (3) to provide a second hydrogen-rich gaseous stream (7) and a liquid stream (9) comprising fuel gas and distillable hydrocarbonaceous compounds; and
 - (c) recovering a distillable hydrocarbonaceous product (12) from said liquid stream (9), and
 - (d) heating (14) at least a portion of said second hydrogen-rich gaseous stream (7) and passing it to step (a) as at least a portion of said hot first hydrogen-rich stream (7).
- 15 2. A process according to claim 1 characterized in that said temperature-sensitive hydrocarbonaceous waste stream (1) comprises a dielectric fluid, hydraulic fluid, heat transfer fluid, used lubricating oil, used cutting oil, used solvent, still bottoms from a solvent recycle operation, coal tar, atmospheric residuum, PCB-contaminated oil, pesticide waste or other hydrocarbonaceous industrial waste, and said non-distillable component comprises organometallic compounds, inorganic metallic compounds, finely divided particulate matter or non-distillable hydrocarbonaceous compounds.
- 20 3. A process according to claim 1 or 2 characterized in that said temperature-sensitive hydrocarbonaceous waste stream (1) is introduced into said flash zone at a temperature less than 482°F (250°C), and wherein the temperature of said hot first hydrogen-rich stream is from 200°F (93°C) to 1200°F (649°C).
- 25 4. A process according to any one of claims 1 to 3 characterized in that said flash conditions include a temperature from 150°F (65°C) to 860°F (460°C), a pressure from atmospheric to 2000 psig (13788 kPa gauge), a hydrogen circulation rate of 1000 SCFB (168 normal m³/m³) to 30,000 SCFB (5056 normal m³/m³) based on said temperature-sensitive hydrocarbonaceous waste stream, and an average residence time of said vapor stream in said flash zone from 0.1 to 50 seconds.
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Patentansprüche

1. Verfahren zur Behandlung eines temperaturempfindlichen kohlenwasserstoffhaltigen Abfallstroms (1), der eine nicht destillierbare Komponente enthält, zur Erzeugung eines destillierbaren kohlenwasserstoffhaltigen Produktes und eines die nicht destillierbare Komponente umfassenden schweren Produktes, während der thermische Abbau dieses kohlenwasserstoffhaltigen Stromes minimiert wird, dadurch gekennzeichnet, daß man:
 - a) den kohlenwasserstoffhaltigen Abfallstrom (1) mit einem heißen ersten wasserstoffreichen Gasstrom (7) mit einer größeren Temperatur als die des kohlenwasserstoffhaltigen Stromes in einer Flash-Zone (2) bei Flash-Bedingungen behandelt und dabei die Temperatur des kohlenwasserstoffhaltigen Stromes erhöht und wenigstens einen Teil desselben verdampft, um einen Wasserstoff und Kohlenwasserstoffe umfassenden Dampfstrom (3) und ein die nicht destillierbare Komponente umfassendes schweres Produkt (6) zu bekommen,
 - b) wenigstens einen Teil des Dampfstromes (3) kondensiert (4), um einen zweiten wasserstoffreichen Gasstrom (7) und einen Trengas und destillierbare kohlenwasserstoffhaltige Verbindungen umfassenden flüssigen Strom (9) zu bekommen,
 - c) aus dem flüssigen Strom (9) ein destillierbares kohlenwasserstoffhaltiges Produkt (12) gewinnt und
 - d) wenigstens einen Teil des zweiten wasserstoffreichen Gasstromes (7) erhitzt (14) und ihn zu der Stufe (a) als wenigstens einen Teil des heißen ersten wasserstoffreichen Stromes (7) führt.
2. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß der temperaturempfindlich kohlenwasserstoffhaltige Abfallstrom (1) ein dielektrisches Fließmittel, hydraulisch s Fließmittel, Wärmeübertragungsfließmittel, verbrauchtes Schmieröl, verbrauchtes Schneidöl, verbrauchtes Lösungsmittel, Destillationsbodenprodukte aus einer Lösungsmittelrückführung, Kohlenteer, atmosphärischen Rückstand, PCB-verunreinigtes Öl, Pestizidabfälle oder andere kohlenwasserstoffhaltige

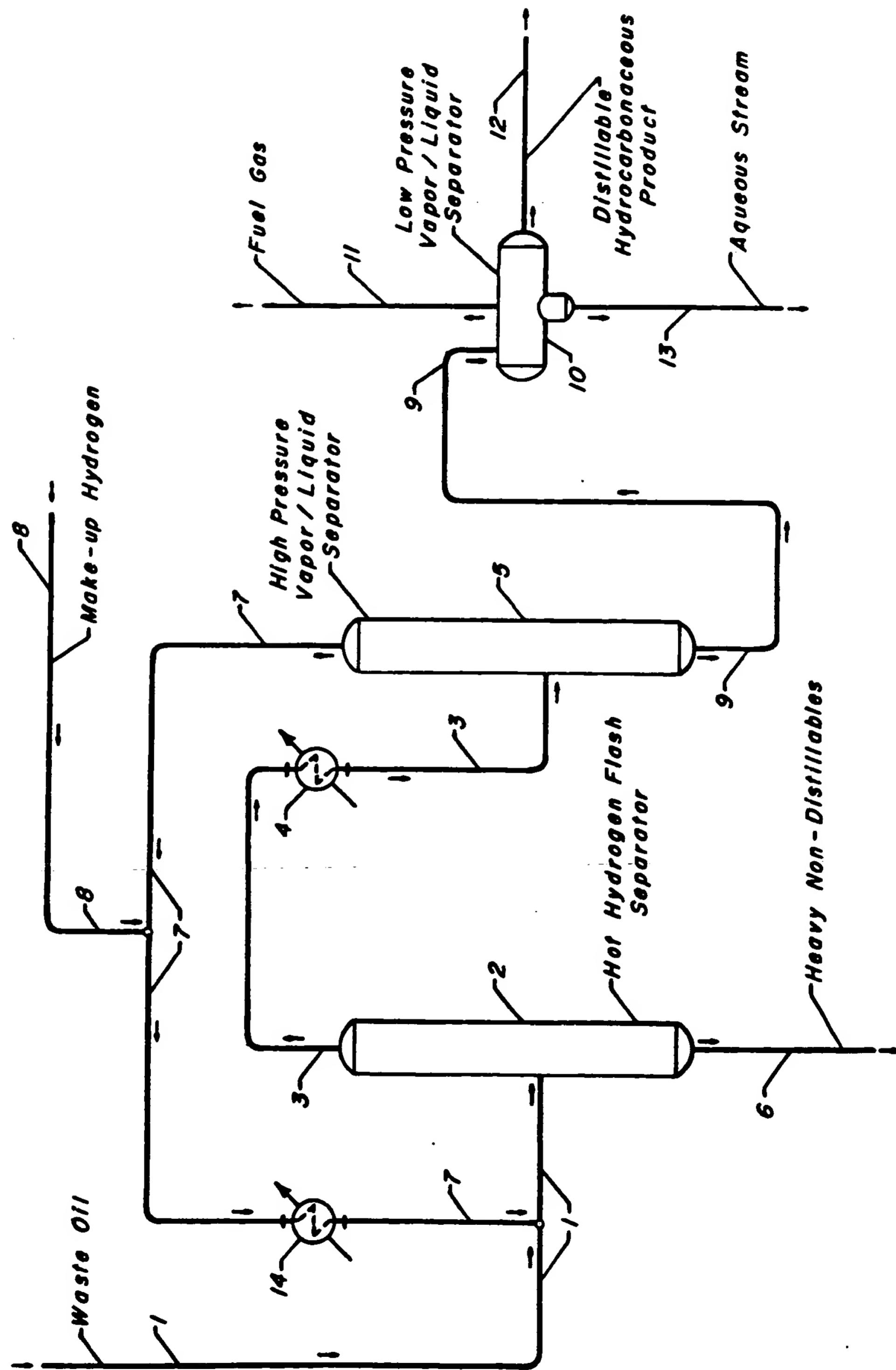
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Industrieabfälle umfaßt und daß die nicht destillierbare Komponente metallorganische Verbindungen, anorganische Metallverbindungen, ferner teile feste Metalle oder nicht destillierbare Kohlenwasserstoffverbindungen umfaßt.

3. Verfahren nach Anspruch 1 oder 2, dadurch gekennzeichnet, daß der temperaturempfindliche kohlenwasserstoffhaltige Abfallstrom (1) in die Flash-Zone bei einer Temperatur geringer als 482°F (250°C) eingeführt wird und die Temperatur des heißen ersten wasserstoffreichen Stromes 200°C (93°C) bis 1200°F (649°C) ist.
4. Verfahren nach einem der Ansprüche 1 bis 3, dadurch gekennzeichnet, daß die Flash-Bedingungen eine Temperatur von 150°F (65°C) bis 860°F (460°C), einen Druck von Atmosphärendruck bis 2000 psig (13788 kPa Überdruck), eine Wasserstoffkreislaufgeschwindigkeit von 1000 SCFB (169 Normal-m³/m³) bis 30 000 SCFB (5056 Normal-m³/m³), bezogen auf den temperaturempfindlichen kohlenwasserstoffhaltigen Abfallstrom, und eine mittlere Verweilzeit des Dampfstromes in der entspannungszone von 0,1 bis sec einschließen.

15 Revendications

1. Procédé pour le traitement d'un courant de déchets hydrocarboné thermosensible (1) contenant un composant non distillable, pour l'obtention d'un produit hydrocarboné distillable et d'un produit lourd comprenant ledit composant non distillable, tout en réduisant au minimum la dégradation thermique dudit courant hydrocarboné, caractérisé par:
 - (a) la mise en contact dudit courant de déchets hydrocarboné (1) avec un premier courant gazeux (7) chaud, riche en hydrogène, ayant une température supérieure à celle dudit courant hydrocarboné, dans une zone de détente (2) dans des conditions de détente, éllevant ainsi la température dudit courant hydrocarboné et vaporisant au moins une partie de celui-ci pour la production d'un courant de vapeur (3) comprenant de l'hydrogène et des hydrocarbures, et d'un produit lourd (6) comprenant ledit composant non distillable;
 - (b) la condensation (4) d'au moins une partie dudit courant de vapeur (3) pour la production d'un second courant gazeux (7) riche en hydrogène et d'un courant liquide (9) comprenant du gaz combustible et des composés hydrocarbonés distillables; et
 - (c) la récupération d'un produit hydrocarboné distillable (12) à partir dudit courant liquide (9), et
 - (d) le chauffage (14) d'au moins une partie dudit second courant gazeux (7) riche en hydrogène et l'envoi de celle-ci à l'étape (a), en tant qu'au moins une partie dudit premier courant (7) chaud, riche en hydrogène.
2. Procédé selon la revendication 1, caractérisé en ce que ledit courant de déchets hydrocarboné thermosensible (1) comprend un fluide diélectrique, un fluide hydraulique, un fluide caloporteur, une huile de graissage usée, une huile de coupe usée, un solvant usé, des résidus de distillation provenant d'une opération de recyclage de solvant, du goudron de houille, un résidu atmosphérique, une huile contaminée par du PCB, des déchets de pesticides ou d'autres déchets industriels hydrocarbonés, et ledit composant non distillable comprend des composés organométalliques, des composés métalliques inorganiques, de la matière particulaire finement-divisée ou des composés hydrocarbonés non distillables.
3. Procédé selon la revendication 1 ou 2, caractérisé en ce que ledit courant (1) de déchets hydrocarboné thermosensible est introduit dans ladite zone de détente à une température inférieure à 250°C (482°F), et dans lequel la température dudit premier courant chaud riche en hydrogène est de 93 à 649°C (200—1 200°F).
4. Procédé selon l'une quelconque des revendications 1 à 3, caractérisé en ce que lesdites conditions de détente comprennent une température de 65 à 460°C (150—860°F), une pression allant de la pression atmosphérique à une pression manométrique de 13 788 kPa (2 000 psi), un débit d'hydrogène de 168 m³ normaux/m³ (1 000 SCFB) à 5 056 m³ normaux/m³ (30 000 SCFB) par rapport audit courant de déchets hydrocarboné thermosensible, et un temps de séjour moyen dudit courant de vapeur dans ladite zone de détente allant de 0,1 à 50 secondes.





(2)

EUROPEAN PATENT APPLICATION

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(5) Int. Cl. C10M 175/00 , C10G 49/22

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A request for addition of a missing page 11 of the originally filed description has been filed pursuant to Rule 88 EPC. A decision on the request will be taken during the proceedings before the Examining Division (Guidelines for Examination in the EPO, A-V, 2.2).

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(5) Treating a temperature-sensitive hydrocarbonaceous waste stream containing a non-distillable component.

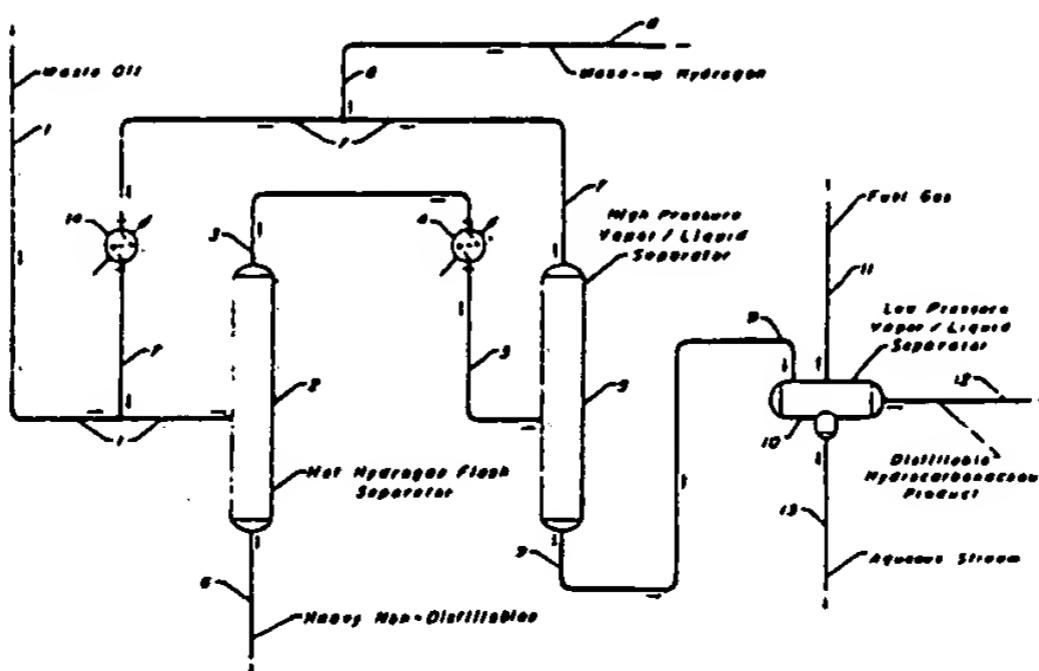
(5) A temperature-sensitive hydrocarbonaceous waste stream (1) containing a non-distillable component is treated to produce a distillable hydrocarbonaceous product and a heavy product comprising the non-distillable component while minimizing thermal degradation of the hydrocarbonaceous stream by a process:

(a) contacting the hydrocarbonaceous waste stream (1) with a hot first hydrogen-rich gaseous stream (7) having a temperature greater than the hydrocarbonaceous stream in a flash zone (2) at flash conditions, thereby increasing the temperature of the hydrocarbonaceous stream and vaporizing at least a portion thereof to provide a vapor stream (3) comprising hydrocarbons and hydrogen and a heavy product (6) comprising the non-distillable component;

(b) condensing (4) at least a portion of the vapor stream (3) to provide a second hydrogen-rich gaseous stream (7) suitable for recycle and a liquid stream (9) comprising fuel gas and distillable hydrocarbonaceous compounds;

(c) recovering a distillable hydrocarbonaceous product (12) from the liquid stream (9), and

(d) heating (14) at least a portion of the second hydrogen-rich gaseous stream (7), and recycling it to form at least a portion of the first hydrogen-rich gaseous stream (7).



TREATING A TEMPERATURE-SENSITIVE HYDROCARBONACEOUS WASTE STREAM CONTAINING A NON-DISTILLABLE COMPONENT

This invention relates to the production of a distillable hydrocarbonaceous product from a temperature-sensitive hydrocarbonaceous waste stream containing a non-distillable component. More specifically, the invention relates to a process using flash vaporization for treating a temperature-sensitive hydrocarbonaceous waste stream containing a non-distillable component to produce a distillable hydrocarbonaceous product and a heavy product comprising a non-distillable component, while minimizing thermal degradation of the hydrocarbonaceous stream.

With the advent of recognition of the dangers associated with disposal of waste streams containing hazardous materials, there has been a steadily increasing demand for technology which is capable of treating a temperature-sensitive hydrocarbonaceous waste stream containing a non-distillable component to produce a distillable hydrocarbonaceous product and a heavy non-distillable product, while minimizing thermal degradation of the hydrocarbonaceous feed stream. Such treatment has always been in demand for the preparation and production of various hydrocarbonaceous products, but with the increased environmental emphasis for the treatment and recycle of waste hydrocarbonaceous products, there is an increased need for improved processes to separate heavy non-distillable components from a distillable hydrocarbonaceous product. For example, during the disposal or recycle of potentially environmentally harmful hydrocarbonaceous waste streams, an important step in the total solution to the problem, is the pretreatment or conditioning of a hydrocarbonaceous stream which facilitates the ultimate resolution to provide product streams which may subsequently be handled in an environmentally acceptable manner. Therefore, those skilled in the art have sought to find feasible techniques to remove heavy non-distillable components from a temperature-sensitive hydrocarbonaceous waste stream to provide a distillable hydrocarbonaceous product. Previous techniques which have been employed include filtration, vacuum wiped-film evaporation, solvent extraction, centrifugation, and vacuum distillation.

The invention provides an improved process for the production of a distillable hydrocarbonaceous product from a temperature-sensitive hydrocarbonaceous waste stream containing a non-distillable component by means of contacting the hydrocarbonaceous waste feed stream with a hot hydrogen-rich gaseous stream under conditions selected to increase the temperature of the feed stream and to flash vaporize at least a portion of the distillable hydrocarbonaceous compounds, thereby producing a distillable hydrocarbonaceous product. Important elements of the improved process are the relatively short time that the feed stream is maintained at elevated temperature, the avoidance of heating the feed stream via indirect heat exchange, and the inhibition of coking reactions which results from the presence of hydrogen.

One embodiment of the invention may be characterized as a process for treating a temperature-sensitive hydrocarbonaceous waste stream containing a non-distillable component to produce a distillable hydrocarbonaceous product and a heavy product comprising the non-distillable component while minimizing thermal degradation of the hydrocarbonaceous stream which process comprises the steps of: (a) contacting the hydrocarbonaceous stream with a hot first hydrogen-rich gaseous stream having a temperature greater than the hydrocarbonaceous stream in a flash zone at flash conditions thereby increasing the temperature of the hydrocarbonaceous stream and vaporizing at least a portion thereof to provide a vapor stream comprising hydrogen and hydrocarbons and a heavy product comprising the non-distillable component; (b) condensing at least a portion of the vapor stream to provide a second hydrogen-rich gaseous stream suitable for recycle and a liquid stream comprising fuel gas and distillable hydrocarbonaceous compounds; and (c) recovering a distillable hydrocarbonaceous product from the liquid stream.

Another embodiment of the invention may be characterized as a process for treating a temperature-sensitive hydrocarbonaceous waste stream containing a non-distillable component to produce a distillable hydrocarbonaceous product and a heavy product comprising the non-distillable component while minimizing thermal degradation of the hydrocarbonaceous stream which process comprises the steps of: (a) contacting the hydrocarbonaceous waste stream with a hot first hydrogen-rich gaseous stream having a temperature greater than the hydrocarbonaceous stream in a flash zone at flash conditions thereby increasing the temperature of the hydrocarbonaceous stream and vaporizing at least a portion thereof to provide a first vapor stream comprising hydrogen and hydrocarbons and a heavy product comprising the non-distillable component; (b) condensing at least a portion of the first vapor stream to provide a second hydrogen-rich gaseous stream suitable for recycle and a liquid stream comprising distillable hydrocarbonaceous compounds and dissolved hydrogen; (c) separating the liquid stream to provide a fuel gas stream and a normally liquid distillable hydrocarbonaceous product, and (d) heating at least a portion of said second hydrogen-rich gaseous stream, and passing it to step (a) as at least a portion of said first hydrogen-rich

gaseous stream.

Other embodiments of the present invention encompass further details such as preferred feedstocks and operating conditions, all of which are hereinafter disclosed in the following discussion of each of these facets of the invention.

5 The drawing is a simplified process flow diagram of a preferred embodiment of the present invention.

The present invention provides an improved process for the removal of heavy non-distillable components from a temperature-sensitive hydrocarbonaceous stream. A wide variety of temperature-sensitive hydrocarbonaceous streams may be employed for feed streams treated in accordance with the process of the present invention. Examples of hydrocarbonaceous streams which are suitable for treatment by the 10 process of the present invention are dielectric fluids, hydraulic fluids, heat transfer fluids, used lubricating oil, used cutting oils, used solvents, still bottoms from solvent recycle operations, coal tars, atmospheric residuum, oils contaminated with polychlorinated biphenyls (PCB), pesticide wastes or other hydrocarbonaceous industrial waste. Many of these hydrocarbonaceous streams may contain non-distillable components which include, for example; organometallic compounds, inorganic metallic compounds, finely divided 15 particulate matter and non-distillable hydrocarbonaceous compounds (i.e. hydrocarbons or hydrocarbonaceous compounds boiling above the maximum boiling point for materials that can be recovered in the overhead from a vacuum fractionation column). The present invention is particularly advantageous when the non-distillable components comprise sub-micron particulate matter and the conventional techniques of filtration or centrifugation tend to be highly ineffective.

20 Once the temperature-sensitive hydrocarbonaceous feed stream is separated into a distillable hydrocarbonaceous product and a heavy non-distillable product, each of these products may be utilized as recovered, or may be subsequently treated or processed by any known technique or process. If the feed stream contains metallic compounds such as those that contain metals such as zinc, copper, iron, barium, phosphorus, magnesium, aluminium, lead, mercury, cadmium, cobalt, arsenic, vanadium, chromium, and 25 nickel, these compounds will be isolated in the relatively small volume of recovered non-distillable product, which may then be treated for metals recovery or otherwise disposed of as desired. In the event that the feed stream contains distillable hydrocarbonaceous compounds which include sulphur, oxygen, nitrogen, metal or halogen components, the resulting recovered distillable hydrocarbonaceous product may be further processed to remove or convert any such components as desired or required.

30 In accordance with the subject invention, a temperature-sensitive hydrocarbonaceous stream containing a non-distillable component is contacted with a hot hydrogen-rich gaseous stream having a temperature greater than the hydrocarbonaceous stream in a flash zone at flash conditions, thereby increasing the temperature of the hydrocarbonaceous stream and vaporizing at least a portion thereof to provide a hydrocarbonaceous vapor stream comprising hydrogen and a heavy non-distillable product. The hydrogen-rich gaseous stream preferably comprises more than 80 mol % hydrogen and more preferably more than 35 90 mol % hydrogen. The hydrogen-rich gaseous stream is multi-functional and serves as 1) a heat source used for directly heating the hydrocarbonaceous feed stream so as to preclude the coke formation that could otherwise occur when using an indirect heating apparatus, such as a heater or heat-exchanger, 2) a diluent to reduce the partial pressure of the hydrocarbonaceous compounds, 3) a possible reactant to 40 minimize the formation of hydrocarbonaceous polymers, and 4) a stripping medium. In accordance with the subject invention, the temperature-sensitive hydrocarbonaceous feed stream is preferably maintained at a temperature less than 482 °F (250 °C) before being introduced into the flash zone, in order to prevent or minimize the thermal degradation of the feed stream. Depending upon the characteristics and composition of the hydrocarbonaceous feed stream, the hot hydrogen-rich gaseous stream is introduced into the flash 45 zone at a temperature greater than the hydrocarbonaceous feed stream and preferably at a temperature from 200 °F (93 °C) to 1200 °F (649 °C).

During the contacting, the flash zone is preferably maintained at flash conditions which include a temperature from 150 °F (65 °C) to 860 °F (460 °C), a pressure from atmospheric to 2000 psig (13788 kPa gauge), a hydrogen circulation rate of 1000 SCFB (168 normal m³/m³) to 30,000 SCFB (5056 normal m³/m³), based on the temperature-sensitive hydrocarbonaceous feed stream, and an average residence 50 time of the hydrogen-containing, hydrocarbonaceous vapor stream in the flash zone from 0.1 to 50 seconds. A more preferred average residence time of the hydrogen-containing, hydrocarbonaceous vapor stream in the flash zone is from 1 to 10 seconds.

The resulting heavy non-distillable portion of the feed stream is removed from the bottom of the flash 55 zone as required to yield a heavy non-distillable product. The heavy non-distillable product may contain a relatively small amount of distillable components but since essentially all of non-distillable components contained in the hydrocarbonaceous feed stream are recovered in this product stream, the term "heavy non-distillable product" is nevertheless used for the convenient description of this product stream. The

heavy non-distillable product preferably contains an atmospheric distillable component of less than 10 weight percent and more preferably less than 5 weight percent. Under certain circumstances, with a feed stream not having an appreciable amount of liquid non-distillable components, it is contemplated that an additional liquid may be utilized to flush the heavy non-distillables from the flash zone. An example of this situation is when the hydrocarbonaceous feed stream comprises a very high percentage of distillable hydrocarbonaceous compounds and relatively small quantities of finely divided particulate matter (solid) and essentially no liquid non-distillable component for use as a carrier for the solids. Such a flush liquid may, for example, be a diesel cut boiling in the range from 500° F (260° C) to 700° F (371° C), a high boiling range vacuum gas oil having a boiling range from 700° F (371° C) to 1000° F (538° C), or a vacuum tower bottoms stream boiling at a temperature greater than 1000° F (538° C). The selection of a flush liquid depends upon the composition of the hydrocarbonaceous feed stream and the prevailing flash conditions in the flash separator, and the volume of the flush liquid is preferably limited to that required for removal of the heavy non-distillable component.

The resulting hydrocarbonaceous vapor stream comprising hydrogen is removed from the flash zone, and at least a portion thereof is condensed to provide a second hydrogen-rich gaseous stream and a liquid stream comprising distillable hydrocarbonaceous compounds.

In the drawing, one embodiment of the subject invention is illustrated by means of a simplified flow diagram, in which such details as pumps, instrumentation, heat-exchange and heat-recovery circuits, compressors and similar hardware have been deleted as being non-essential to an understanding of the techniques involved. The use of such miscellaneous apparatus is well known to one skilled in the art of hydrocarbon processing techniques. With reference now to the drawing, a waste oil feed stream having a non-distillable component is introduced into the process via conduit (1), and is contacted with a hot gaseous hydrogen-rich recycle stream which is provided via conduit (7) and hereinafter described. The waste oil and the hydrogen-rich recycle stream are intimately contacted in hot hydrogen flash separator (2). A hydrocarbonaceous vapor stream comprising hydrogen is removed from hot hydrogen flash separator (2) via conduit (3), cooled in heat-exchanger (4), and introduced into high pressure vapor/liquid separator (5). A heavy non-distillable stream is removed from the bottom of hot hydrogen flash separator (2) via conduit (6) and recovered. A hydrogen-rich gaseous stream is removed from separator (5) via conduit (7), heated to a suitable temperature in heat-exchanger (14), and utilized to contact the waste oil feed stream and hereinabove described. Since hydrogen is lost from the process because a portion of the hydrogen will be dissolved in the liquid hydrocarbon leaving separator (5), it is necessary to supplant the lost hydrogen with make-up hydrogen from some suitable external source, i.e., a catalytic reforming unit or a hydrogen plant. Make-up hydrogen may be introduced into the system at any convenient and suitable point, and is introduced in the embodiment shown in the drawing via conduit (8). A liquid hydrocarbonaceous stream containing hydrogen in solution is removed from high pressure vapor/liquid separator (5) via conduit (9) and is introduced into low pressure vapor/liquid separator (10). A gaseous stream comprising hydrogen and any normally gaseous hydrocarbons present is removed from low pressure vapor/liquid separator (10) via conduit (11) and recovered. A normally liquid distillable hydrocarbonaceous product is removed from low pressure vapor/liquid separator (10) via conduit (12) and recovered. In the event that the waste oil feed stream contains water, this water is recovered from low pressure vapor/liquid separator (10) via conduit (13).

The following Example is presented for the purpose of further illustrating the process of the present invention, and to indicate the benefits afforded by the utilization thereof in producing a distillable hydrocarbonaceous product, while minimizing thermal degradation of the temperature-sensitive hydrocarbonaceous feed stream containing a non-distillable component.

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EXAMPLE

50 A waste lube oil containing fully divided particulate matter and heavy metals having the characteristics presented in Table 1, and contaminated with 1020 parts per million by weight (wppm) of polychlorinated biphenyl (PCB), in the form of Aroclor, was charged at a rate of 100 mass units per hour to a hot hydrogen flash zone. The hot hydrogen was introduced into the hot hydrogen flash zone at a rate of 31 mass units per hour.

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TABLE 1

| WASTE LUBE OIL FEEDSTOCK PROPERTIES (5375-45) | | | |
|---|--|-------|--------|
| 5 | Specific Gravity @ 60° F (15° C) | .8827 | |
| 10 | Vacuum Distillation Boiling Range, (ASTM D-1160) | ° F | (° C) |
| 15 | IBP | 338 | (170) |
| 20 | 10% | 516 | (269) |
| | 20% | 628 | (331) |
| | 30% | 690 | (367) |
| | 40% | 730 | (388) |
| | 50% | 750 | (399) |
| | 60% | 800 | (421) |
| | 70% | 831 | (444) |
| | 80% | 882 | (474) |
| 25 | % Over | 80 | |
| | % Bottoms | 20 | |
| 30 | Sulphur, weight percent | | 0.5 |
| | Polychlorinated Biphenyl Concentration, wppm | | 1020 |
| | Lead, wppm | | 863 |
| | Zinc, wppm | | 416 |
| | Cadmium, wppm | | 1 |
| | Copper, wppm | | 21 |
| | Chromium, wppm | | 5 |

The waste lube oil was preheated to a temperature of <482° F (<250° C) before introduction into the flash zone, which temperature precluded any significant detectable thermal degradation. The waste lube oil was intimately contacted in the flash zone with a hot hydrogen-rich gaseous stream having a temperature upon introduction into the flash zone of >748° F (>398° C). In addition, the hot hydrogen flash zone was operated at conditions which included a temperature of 748° F (398° C), a pressure of 500 psig (3447 kPa gauge), a hydrogen circulation rate of 18000 SCFB (3034 normal m³·m³) and an average residence time of the vapor stream of 5 seconds. A hydrocarbonaceous vapor stream comprising hydrogen was recovered from the flash separation zone, cooled to 77° F (25° C) and introduced into a high pressure separator. An overhead gas stream containing 100% hydrogen in an amount of 31 mass units per hour was recovered from the high pressure separator and found to be suitable for recycle to the flash zone. A liquid stream was removed from the high pressure separator and introduced into a low pressure separator to provide a fuel gas stream containing hydrogen and a small amount of light hydrocarbons and a liquid bottoms stream in the amount of 88 mass units per hour having the characteristics presented in Table 2.

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TABLE 2

| ANALYSIS OF LOW PRESSURE SEPARATOR BOTTOMS STREAM | | | |
|---|--|-------|--------|
| 5 | Specific Gravity @ 60° F (15° C) | 0.866 | |
| 10 | Vacuum Distillation Boiling Range. (ASTM D-1160) | ° F | (° C) |
| 15 | IBP | 225 | (107) |
| 20 | 10% | 433 | (223) |
| 25 | 20% | 538 | (280) |
| 30 | 30% | 633 | (334) |
| 35 | 40% | 702 | (372) |
| 40 | 50% | 741 | (394) |
| 45 | 60% | 770 | (410) |
| 50 | 70% | 801 | (427) |
| 55 | 80% | 837 | (447) |
| 60 | 90% | 896 | (479) |
| 65 | 95% | 943 | (506) |
| 70 | EP | 982 | (527) |
| 75 | % Over | 97 | |
| 80 | % Bottoms | 3 | |
| 85 | Sulphur, weight percent | | 0.31 |
| 90 | Polychlorinated Biphenyl Concentration, wppm | | 1143 |
| 95 | Lead, wppm | | 3.7 |
| 100 | Zinc, wppm | | 1.5 |
| 105 | Cadmium, wppm | | <0.04 |
| 110 | Copper, wppm | | 0.1 |
| 115 | Chromium, wppm | | 0.6 |

A non-distillable liquid stream was recovered from the bottom of the flash separation zone in an amount of 12 mass units per hour and having the characteristics presented in Table 3.

35 TABLE 3 - ANALYSIS OF NON-DISTILLABLE STREAM

Specific Gravity @ 60° F (15° C) 0.9

40 Polychlorinated Biphenyl Concentration, wppm 110

In summary, this example demonstrated that a waste lube oil having a non-distillable component and containing 1020 wppm of polychlorinated biphenyl and 1306 wppm of heavy metals, i.e., lead, zinc, cadmium, copper and chromium, was separated into a distillable hydrocarbonaceous stream containing 98.6 weight percent of the polychlorinated biphenyl contained in the waste lube oil and a heavy stream comprising essentially all of the non-distillable component of the waste lube oil, including 99.5 weight percent of the heavy metals. The analysis of the overhead gas stream showed that the temperature-sensitive waste lube oil did not experience any significant amounts of undesirable thermal cracking, with the accompanying formation of normally gaseous hydrocarbonaceous compounds.

50 Claims

- 55 1. A process for treating a temperature-sensitive hydrocarbonaceous waste stream (1) containing a non-distillable component to produce a distillable hydrocarbonaceous product, and a heavy product comprising said non-distillable component while minimizing thermal degradation of said hydrocarbonaceous stream characterized by:

(a) contacting said hydrocarbonaceous waste stream (1) with a hot first hydrogen-rich gaseous stream (7) having a temperature greater than said hydrocarbonaceous stream, in a flash zone (2) at flash conditions, thereby increasing the temperature of said hydrocarbonaceous stream and vaporizing at least a portion thereof to provide a vapor stream (3) comprising hydrogen and hydrocarbons, and a heavy product (6) comprising said non-distillable component;

(b) condensing (4) at least a portion of said vapor stream (3) to provide a second hydrogen-rich gaseous stream (7) and a liquid stream (9) comprising fuel gas and distillable hydrocarbonaceous compounds; and

(c) recovering a distillable hydrocarbonaceous product (12) from said liquid stream (9), and
 10 (d) heating (14) at least a portion of said second hydrogen-rich gaseous stream (7) and passing it to step (a) as at least a portion of said hot first hydrogen-rich stream (7).

2. A process according to claim 1 characterized in that said temperature-sensitive hydrocarbonaceous waste stream (1) comprises a dielectric fluid, hydraulic fluid, heat transfer fluid, used lubricating oil, used cutting oil, used solvent, still bottoms from a solvent recycle operation, coal tar, atmospheric residuum, 15 PCB-contaminated oil, pesticide waste or other hydrocarbonaceous industrial waste, and said non-distillable component comprises organometallic compounds, inorganic metallic compounds, finely divided particulate matter or non-distillable hydrocarbonaceous compounds.

3. A process according to claim 1 or 2 characterized in that said temperature-sensitive hydrocarbonaceous waste stream (1) is introduced into said flash zone at a temperature less than 482 °F (250 °C), 20 and wherein the temperature of said hot first hydrogen-rich stream is from 200 °F (93 °C) to 1200 °F (649 °C).

4. A process according to any one of claims 1 to 3 characterized in that said flash conditions include a temperature from 150 °F (65 °C) to 860 °F (460 °C), a pressure from atmospheric to 2000 psig (13788 kPa gauge), a hydrogen circulation rate of 1000 SCFB (168 normal m³/m³) to 30,000 SCFB (5056 normal m³ m³) 25 based on said temperature-sensitive hydrocarbonaceous waste stream, and an average residence time of said vapor stream in said flash zone from 0.1 to 50 seconds.

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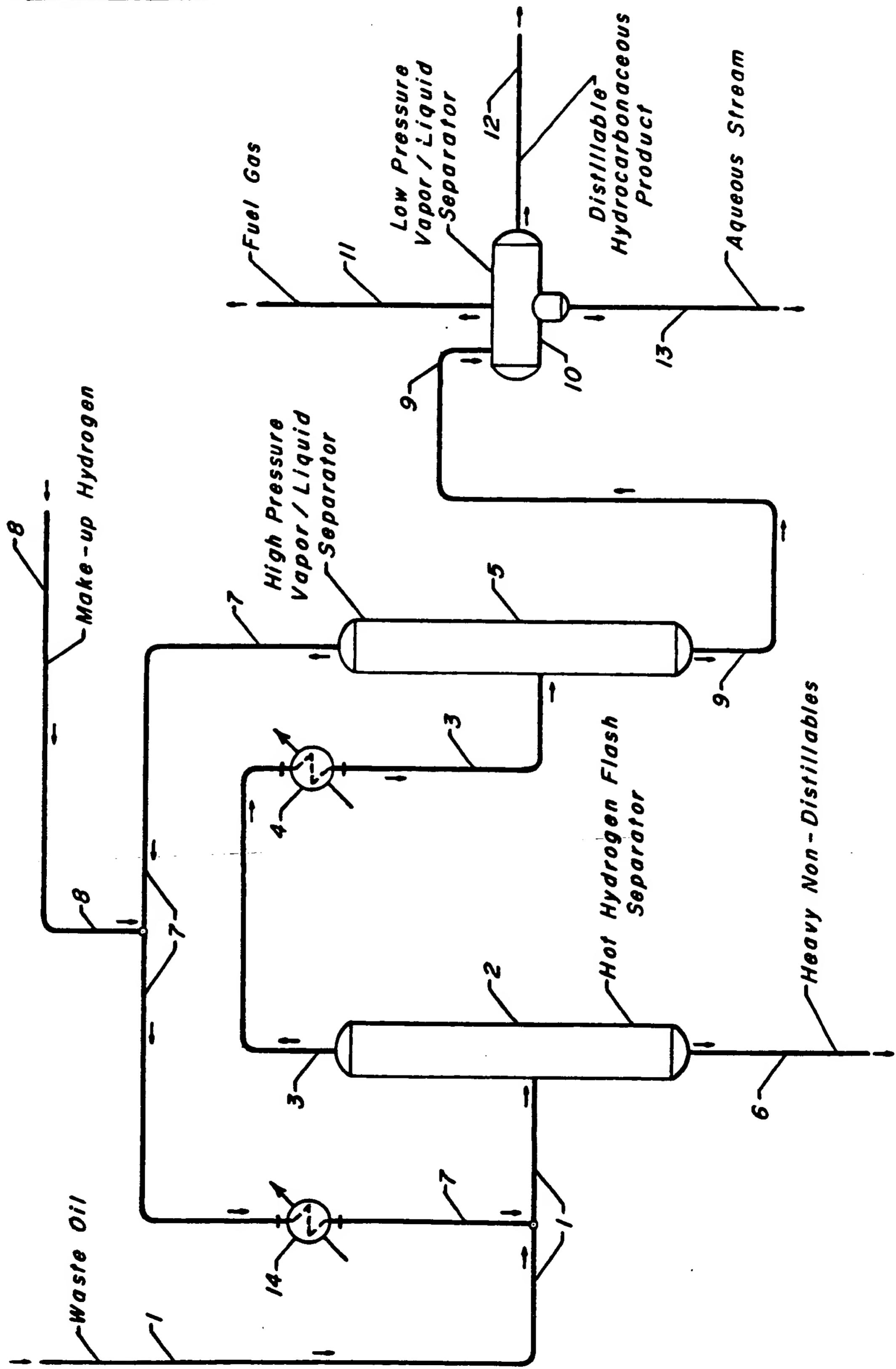
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Neu eingereicht / Newly filed
Nouvellement déposé





| DOCUMENTS CONSIDERED TO BE RELEVANT | | | CLASSIFICATION OF THE APPLICATION (Int. Cl. 4) |
|--|--|---|--|
| Category | Citation of document with indication, where appropriate, of relevant passages | Relevant to claim | |
| A | EP-A-0 149 862 (KINETICS TECHNOLOGY INTERNATIONAL B.V.) --- | 2 | C 10 M 175/00 C 10 G 49/22 |
| A | DE-B-1 182 377 (H.J. KETTLITZ MINERALOEL-RAFFINERIE) * Column 1, line 1 - column 2, line 50 * --- | 1-4 | |
| A | EP-A-0 228 651 (UNION RHEINISCHE BRAUNKOHLENKRAFTSTOFF AG) * Page 1, line 1 - page 4, line 43 * --- | 1-4 | |
| A | US-A-4 265 733 (D.C. TABLER et al.) * Column 4, line 14 - column 6, line 62 * --- | 1-4 | |
| A | US-A-4 151 072 (G.P. NOWACK et al.) * Abstract; figures 1,2; column 2, line 57 - column 3, line 39; column 5, line 36 - column 14, line 58 * ----- | 1-4 | TECHNICAL FIELDS SEARCHED (Int. Cl. 4) |
| | | | C 10 M |
| The present search report has been drawn up for all claims | | | |
| Place of search | Date of completion of the search | Examiner | |
| THE HAGUE | 30-11-1988 | FISCHER W.H.F. | |
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